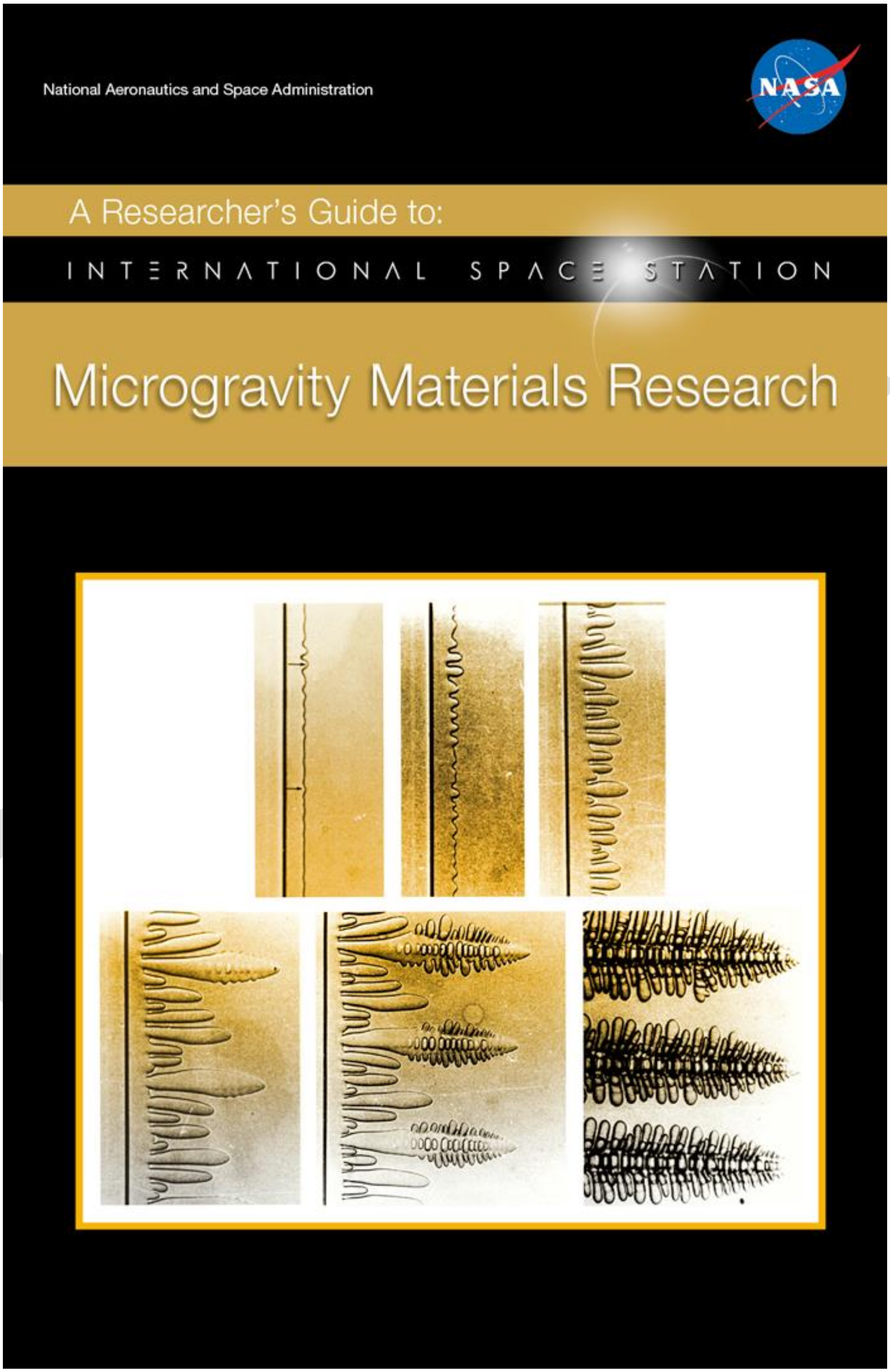
MICROGRAVITY MATERIALS RESEARCH

***** DRAFT *****



WHY USE ISS AS A LABORATORY FOR MATERIALS RESEARCH

Most materials are formed from a partially or totally fluid sample and the transport of heat and mass inherently influences the formation of the material and its resultant properties. The International Space Station (ISS) provides a long-duration spaceflight environment for conducting microgravity materials experiments. The microgravity environment greatly reduces buoyancy-driven convection, pressure head and sedimentation in fluids. The reduction in these gravity-related sources of heat and mass transport may be used to determine how the material processes are affected by gravitational-driven and gravitationally independent sources of heat and mass transfer.

MICROGRAVITY ACCELERATION ENVIRONMENT

The ISS is in orbit, also called free fall, around Earth. The force of Earth's gravity on the ISS is not much less than the force ISS would experience if it were on Earth's surface. Intuitively, it would seem that the orbital experimental environment would not be much different than on Earth's surface either.

However, there is an important experimental difference. On the ground, the experiment containers are fixed to the surface of the Earth and do not move even though they are experiencing one g of force. The fluids in the containers are free to move within their containers and deform based on the force of gravity with the more dense material sinking to the bottom and displacing the less dense material to the top. On ISS, the entire space station including the experiment containers and the fluids within are all moving because of gravity. The differences between the acceleration of the ISS, its experiment containers, and the samples within are quite small. As a result, there is very little sedimentation or buoyancy-driven convection occurring within fluid bodies during ISS experiments. The small difference in acceleration between the orbiting sample and the ISS equipment holding the sample typically averages on the order of one millionth of a g, one microgravity of acceleration. This is the origin of the term "microgravity environment." It is critical for investigators to remember that the effective ISS environment on experiment samples is not zero g. Most microgravity materials investigations require that any sedimentation velocity or density-driven fluid velocity be much smaller than diffusive transport within a length scale of interest. The following sections provide a short description of the accelerations experienced relative to the laboratory frame of reference, i.e., the space station.

QUASI-STEADY ACCELERATIONS

One source of acceleration is drag from the upper atmosphere, and this drag is typically on the order of less than 10^{-7}g's. The drag decelerates ISS and anything firmly attached to it. The fluid samples are not firmly attached and therefore begin to deform as they would on the ground, though to a much less degree, with the less dense material moving towards the direction of the drag acceleration on ISS relative to the more dense material. Another, larger source of acceleration affecting ISS experiment samples is the so-called gravity gradient. The acceleration of gravity vector, **g**, on an Earth-orbiting object varies according to:

$$\mathbf{g} = (GM/R^3)\mathbf{R}$$

This is where G is the gravitational constant, M is the mass of the Earth, and **R** is the vector between the object and the center of the Earth. From the equation, it is clear that **g** varies with orbital position, and it is from this equation that the term gravity gradient, d**g**/d**R**, is derived. At the ISS orbit, the value of **g** varies about 0.3micro-gs/meter along the direction of **R**. It also varies by about 0.1 microg/meter along the axis perpendicular to both **R** and the orbital path of ISS. The direction of the movement of ISS along this path is known as the velocity vector. The effect of gravity gradient can be imagined easily. If an experiment is above the ISS center of mass (larger **R**), then it is farther from Earth and is not accelerated as much by Earth's gravity as ISS. Since the experiment container is firmly attached to ISS, this implies ISS must exert an additional small acceleration on the container so that it travels with ISS. The fluid sample in the container feels Earth's acceleration just like the container, but since the fluid is not rigidly attached to ISS, it deforms because of the slightly different acceleration between itself and the ISS/container. Similarly, if an experiment container is located to the side of the orbital plane of the ISS orbit, then the experiment container would tend to drift across the plane of the orbit of

ISS if the ISS did not exert acceleration on the experiment container to keep it fixed in place relative to ISS. The above accelerations can typically be considered as steady for the purposes of most experiments. Other accelerations occurring on ISS have more time-dependent aspects.

PERIODIC ACCELERATION

ISS has a number of sources of vibrations such as flexing of the structures of ISS, movements of astronauts particularly during exercise, the motions of equipment, etc. The acceleration that is due to oscillations is somewhat more complex for an investigator to analyze. At low-frequency oscillations, a fluid may behave as if it is subjected to a quasi-static force. Alternatively, high-frequency oscillations would exert little influence on fluid motions since viscosity prevents the fluid from developing significant motion during the short duration of time between the application of acceleration in one direction and the subsequent reversal that is due to the periodic nature of an oscillatory acceleration. The transition between the behavior at low- and high-frequency accelerations is gradual and depends on the properties of the fluid and the length scale of interest. This transition occurs when the value of a diffusive property of the fluid is about equal to the angular frequency of the vibration, ω, times the relevant length scale squared, i.e., $D = \omega L^2$.

Periodic oscillations have the effect of adding to the apparent dissipation of momentum, heat and species. The additional effect of a periodic oscillation on the apparent value of these quantities can be approximated by the following:

$v_{eff} = [v^2+(\omega L^2)^2]^{1/2}$

$\alpha_{eff} = [\alpha^2+(\omega L^2)^2]^{1/2}$

$D_{eff} = [D^2+(\omega L^2)^2]^{1/2}$

In these examples, v, α, and D are the kinematic viscosity, thermal diffusivity, and molecular diffusivity respectively. A good review of this subject is Chapter 17 of Fluid Sciences and Material Science in Space, editor H.U. Walter, published by Springer Berlin (1987).

TRANSIENT ACCELERATIONS

Other vehicles visit the ISS and in these docking or berthing events, the ISS velocity is altered. An even larger change in velocity occurs when the orbit of the ISS is adjusted. Unlike the vibrational accelerations discussed above, there is no periodicity to these acceleration events. The resultant disturbance on a fluid body attached to ISS is typically analyzed on the product of acceleration times the duration, i.e., the change in velocity. During these events, the less dense regions of fluid will accelerate in the direction of the change in velocity relative to the more dense regions of fluid. Microgravity investigations are not typically run during these events to avoid the impacts associated with them.

ACCELERATION EXAMPLES

The ISS has accelerometers, which record the acceleration environment and models to predict the accelerations experienced based on the various motors, machinery and activities occurring on ISS. The accelerations can be displayed in any of a number of manners depending on the details of greatest interest. One common format is to use a Fourier transform to show the accelerations as a function of frequency rather than time. The following are some plots showing examples of the ISS environment in some of the formats used most frequently.

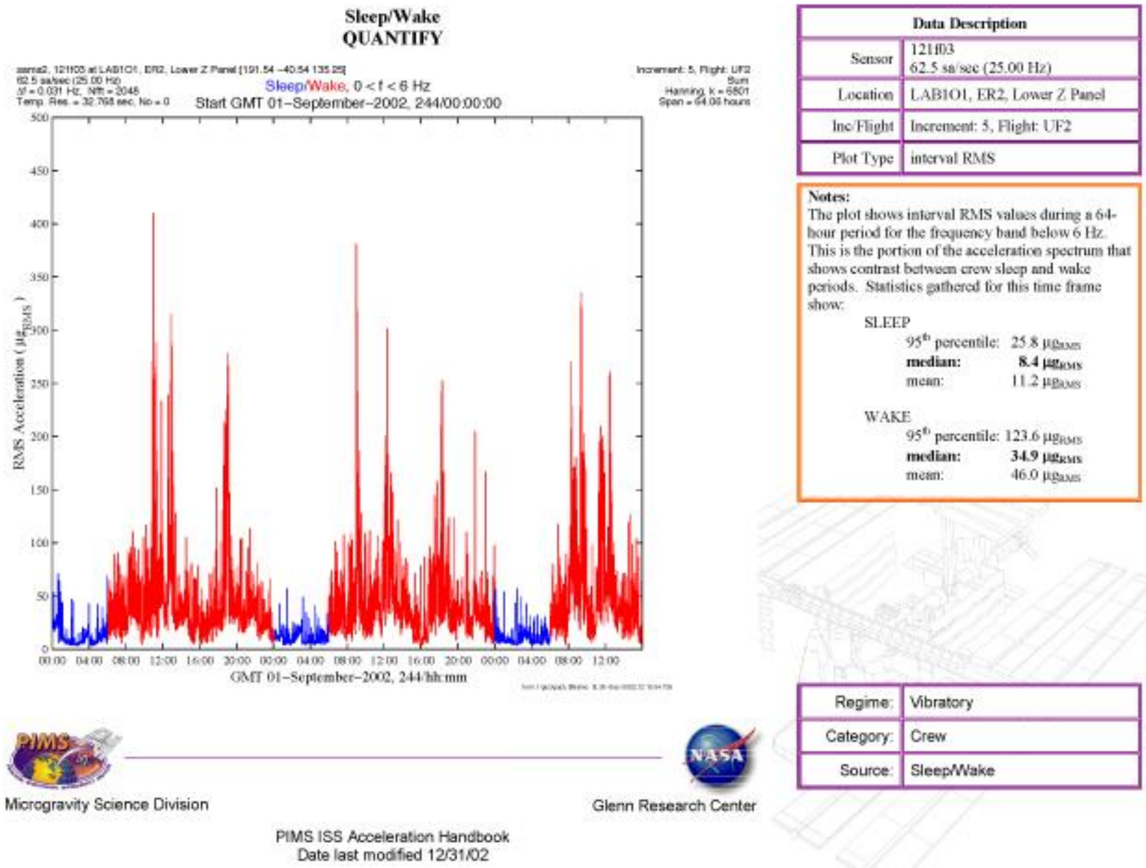


Figure 1: Root Mean Square depiction of the ISS laboratory environment as a function of time. The lowest accelerations occur during the crew sleep periods colored in blue in the figure.

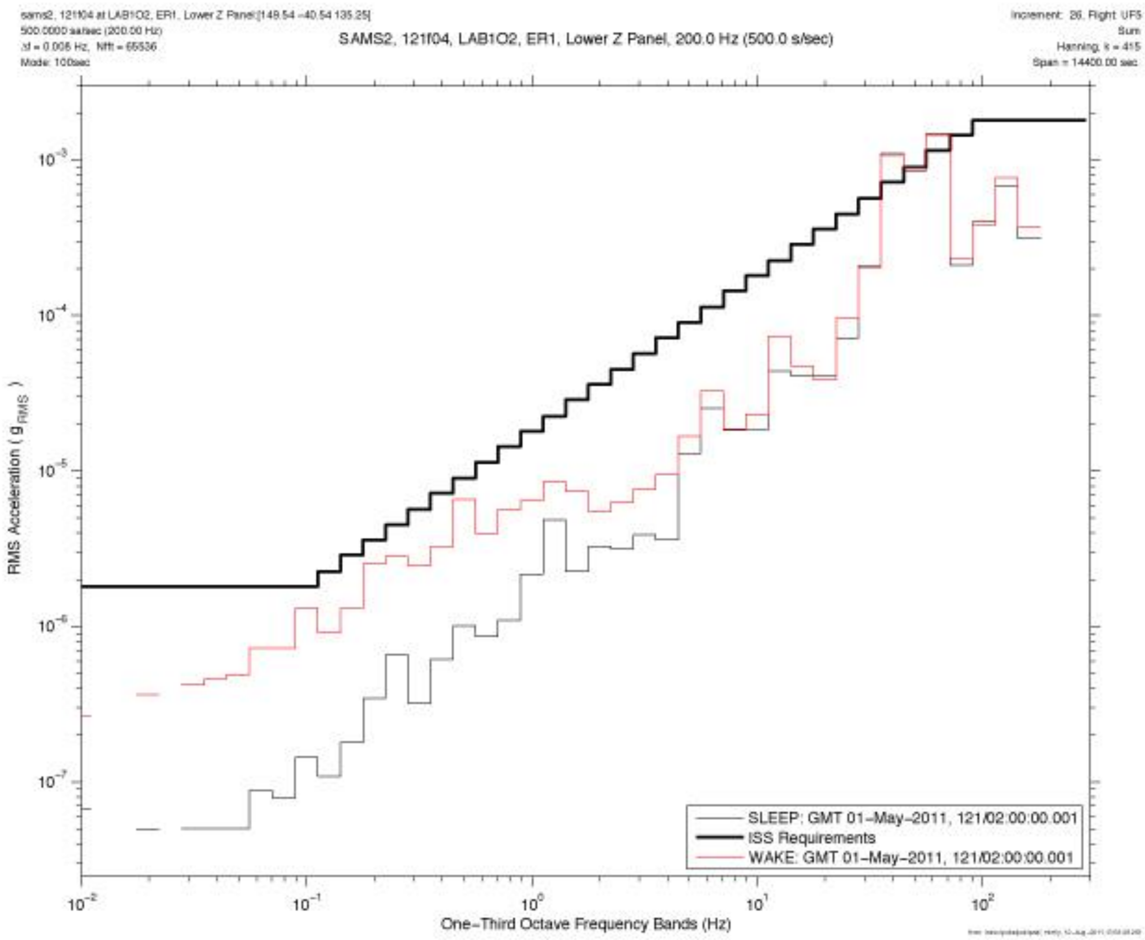


Figure 2: Root Mean Square plot of the accelerations as a function of frequency during crew sleep (thin black line) and during the crew active time frame (red line). The thick black line is the ISS maximum microgravity requirement. Note that this requirement only applies to activate Acceleration Isolated Racks during specifically scheduled periods and does not include the acceleration frequently needed to re-center the floating part of the rack volume.

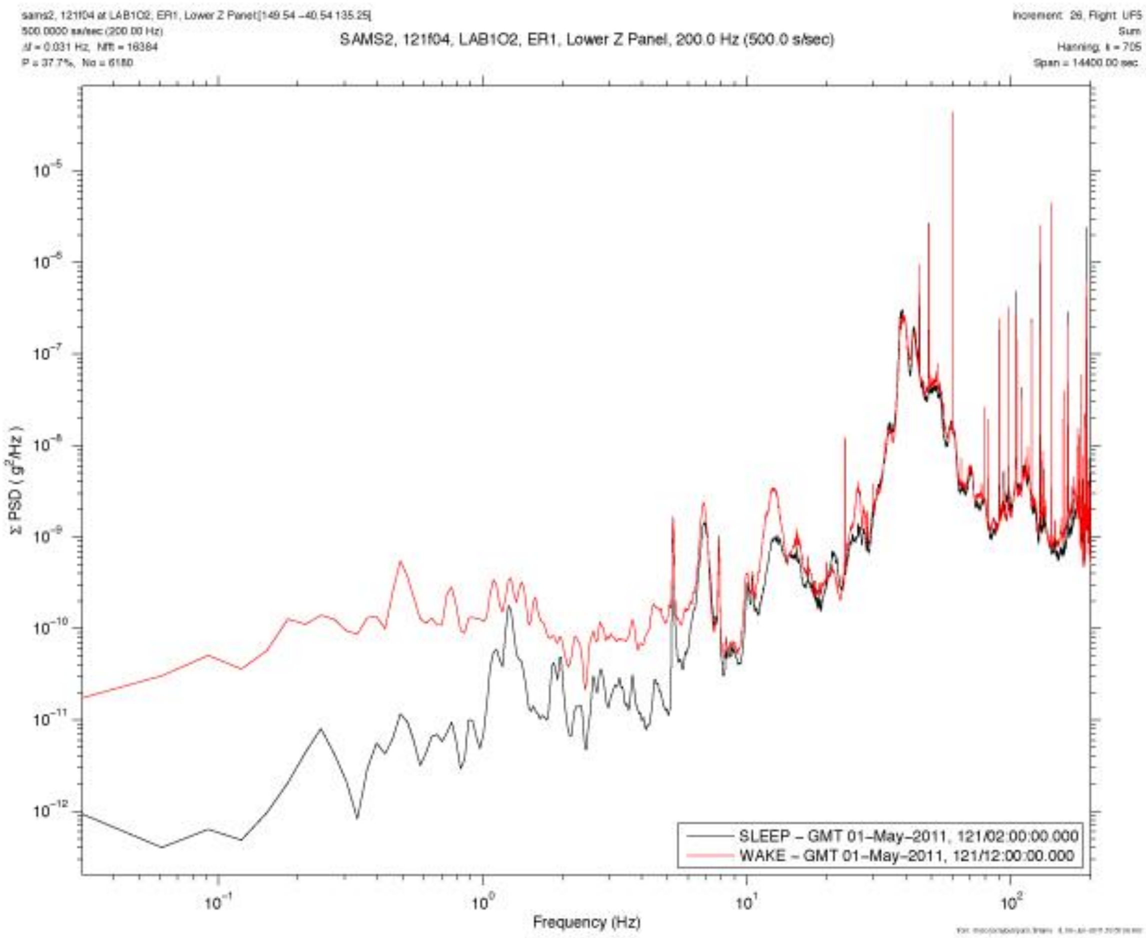


Figure 3: The power spectral density of the accelerations as a function of frequency as a function of crew wake (red line) and sleep (black line) periods.

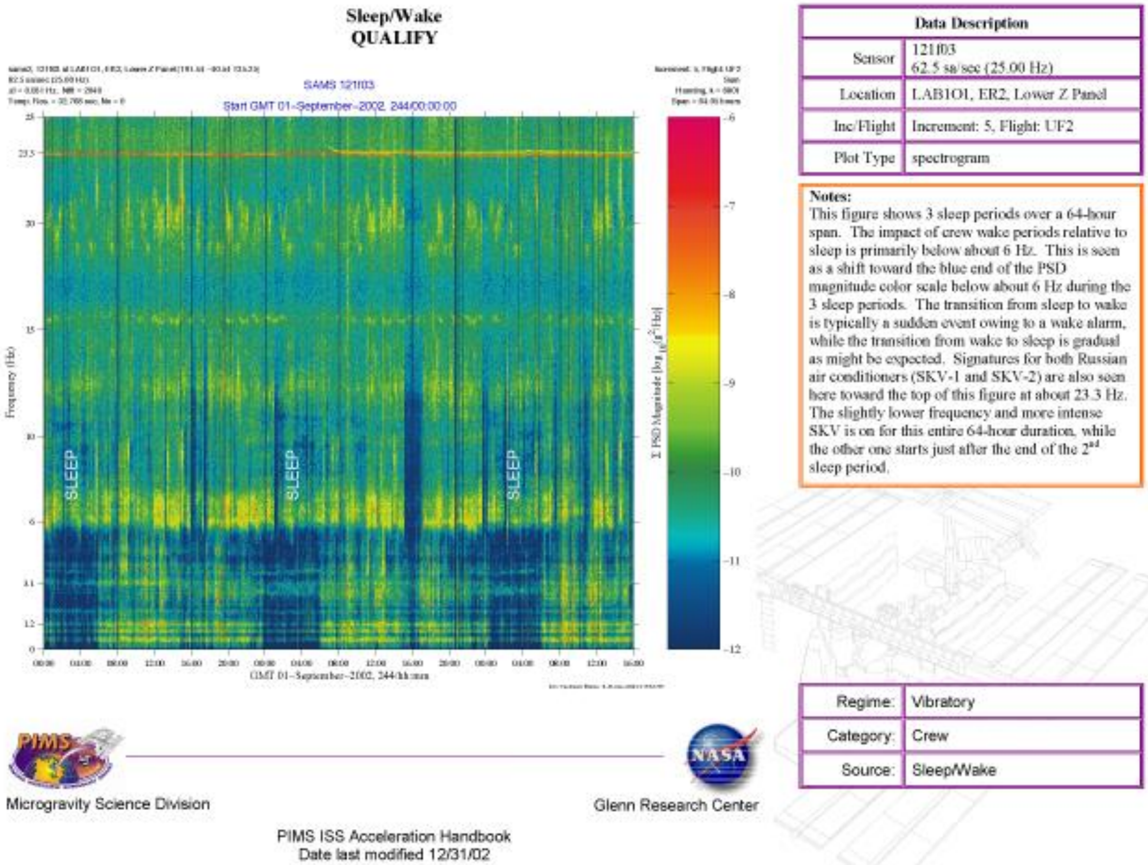


Figure 4: Power spectral density (left axis) plotted as a function of time (bottom axis) and frequency (color with the scale shown on the right). Less intense disturbances are seen during the crew sleep periods.

FACILITIES FOR MATERIALS RESEARCH

LOGISTICS, OPERATIONS, and ACCOMMODATIONS OF FLIGHT EXPERIMENTS

Logistics vehicles deliver the experiments to ISS. These experiments are performed on ISS using some combination of crew control, autonomous pre-programmed operations, or ground commanding of the hardware. Most of the payloads carried by the logistics vehicles are in an ambient environment, but refrigerators and freezers are available for some temperature-sensitive payloads.

MATERIALS SCIENCE RESEARCH RACK (MSRR)

The Materials Science Research Rack (MSRR) is a research facility developed under a cooperative research agreement between NASA and European Space Agency (ESA) for materials science investigations on the ISS.

The MSRR accommodates advanced investigations in the microgravity environment of the ISS for basic materials science research in areas such as solidification of metals and alloys. The purpose is to advance the scientific understanding of materials processing as affected by microgravity and to gain insight into the physical behavior of materials processing. MSRR allows for the study of a variety of materials including metals, ceramics, semiconductor crystals and glasses.

MSRR is a highly automated facility with a modular design capable of supporting multiple types of investigations. Currently, the NASA-provided Rack Support Subsystem provides services (power, thermal control, vacuum access, and command and data handling) to the ESA-developed Materials Science Laboratory (MSL), which accommodates interchangeable Furnace Inserts (FI). Two ESA-developed FIs are presently available on the ISS and can be changed out in orbit: the Low Gradient Furnace (LGF) and the Solidification and Quenching Furnace (SQF). The LGF is designed to achieve a well-controlled low or medium thermal gradient inside the sample between one high-temperature and one low-temperature heater zone, with an adiabatic zone between the two. The SQF is a Bridgman furnace designed to provide for high gradients typically in the 50 to 150-K/cm range inside the sample cartridge. The SQF consists of one hot cavity, an exchangeable adiabatic zone, and water-cooled chill block acting as a heat sink. Sample-Cartridge Assemblies (SCAs), each containing one or more material samples, are installed in the FI by the crew and can be processed at temperatures up to 1,400°C. Once an SCA is installed, the experiment can be run by automatic command or science conducted via telemetry commands from the ground.

This facility is available to support additional materials science investigations through programs such as the U.S. National Laboratory, Technology Development, NASA Research Announcements and others.

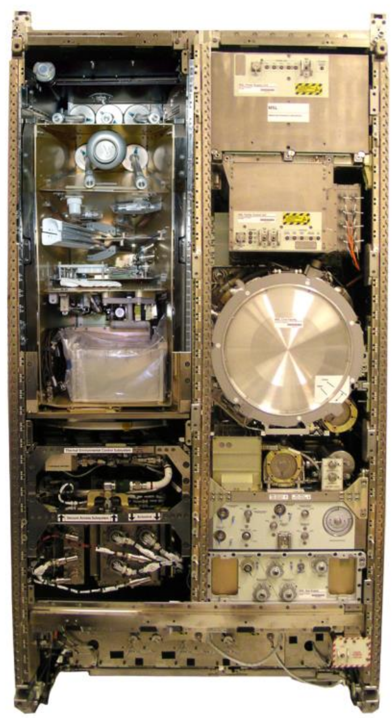


Figure 5: Materials Science Research Rack in-orbit configuration.

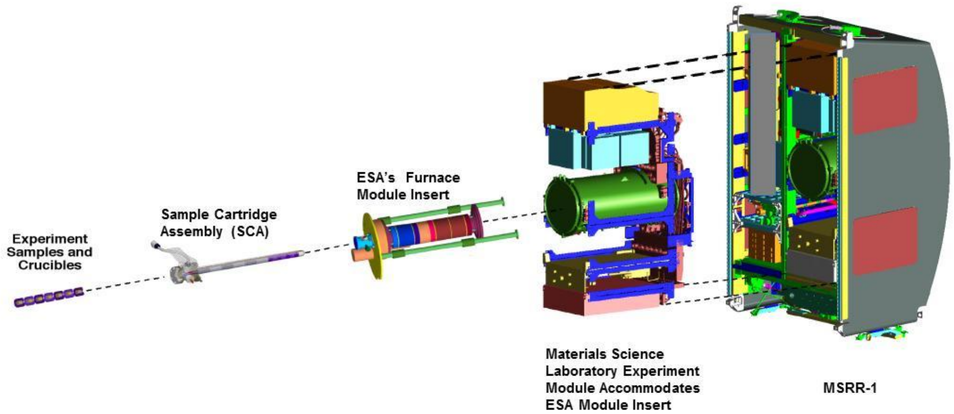


Figure 6: Materials Science Laboratory integration into the Materials Science Research Rack.

MICROGRAVITY SCIENCE GLOVEBOX HARDWARE

The Microgravity Science Glovebox (MSG), also housed in the U.S. Laboratory, Destiny, enables scientists from multiple disciplines to participate actively in the assembly and operation of experiments in space with much the same degree of involvement they have in their own research laboratories. Developed by ESA and managed by NASA's Marshall Space Flight Center (MSFC), the MSG was launched on the Space Shuttle *Endeavor*, STS-111, ISS Flight UF2, in June 2002. The MSG facility offers an enclosed 255-liter (9-cubic-foot) work area accessible to the crew through glove ports and to ground-based scientists through real-time data links and video. Because the work area is sealed and held at a negative pressure, the crew can manipulate experiment hardware and samples without the danger of small parts, particulates, fluids, gasses, or biological material escaping into the open laboratory module.

An airlock under the Work Volume (WV) can be accessed to bring objects in safely while other activities are going on inside MSG. The MSG has 40-cm diameter side ports (equipped with rugged gloves that are sealed to prevent leaks) for setting up and manipulating equipment in the WV. A coldplate provides cooling for experiment hardware, and the air is continuously circulated and filtered. Experiments are provided with 1 Kw of power and cooling.

Vacuum, venting, nitrogen gas input (that can keep the oxygen volume at 10 percent or less), power and data interfaces are also provided within MSG. A video system consists of a self-standing subsystem of four color cameras, two monitors, two analog recorders and two digital recorders integrated into an International Subrack Interface Standard drawer. The command and monitoring panel monitors the facility status and performance and provides for manual operation of MSG by the crew.

In order to support life science research, MSG provides disposable exam gloves and specialized filters for handling typical life science materials. MSG also has an ultraviolet LED decontamination system. The MSG accommodates small and medium-sized investigations from any disciplines including biotechnology, combustion science, life sciences, fluid physics, fundamental physics and materials science. Many of these experiments use chemicals, burning or molten materials or other hazards that must be contained.

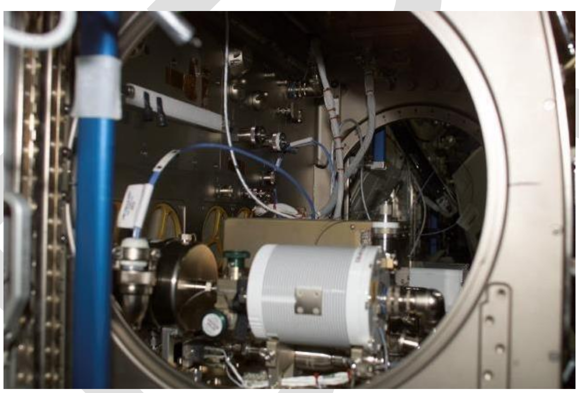
Need captions for these images.

The Coarsening of Solid-Liquid Mixtures (CSLM) project mentioned previously is one example of hardware flown in the MSG. It has been successfully operated on ISS since 2003 with five separate launches of 22 Sample Processing Units with the most recent occurring as a Sortie; launching on SpaceX-2 and returning 30 days later in the SpaceX-2 Dragon capsule. The ability to launch, process the samples, and return the hardware in less than two months significantly increases the quality of the data the Principle Investigator (PI) is able to obtain upon inspection of the processed samples.

CSLM consists of an Electronics Control Unit (ECU), which functions as the Data Acquisition & Controller and the Sample Processing Units (SPUs) that house the samples to be processed. The ECU has remained on ISS for the last 10 years. Briefly, the function of the ECU is to control the heaters in the SPU and maintain the required temperature up to185 °C for a specific period of time at which point the ECU commands the SPU to quench cool the samples. During the entire time, the ECU is recording the temperature data from the four RTDs embedded in the SPU. Data is transferred to the MSG laptop and downlinked to the ground.

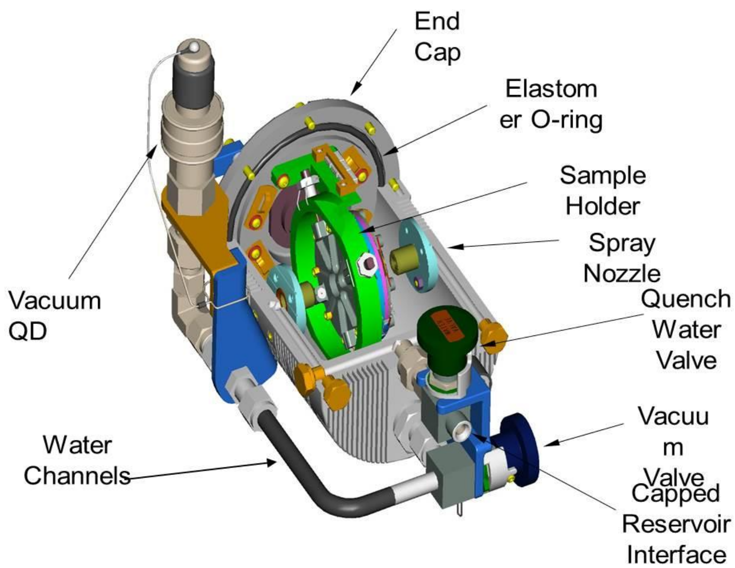
CSLM Sample Processing Unit

CSLM Electronics Control Unit

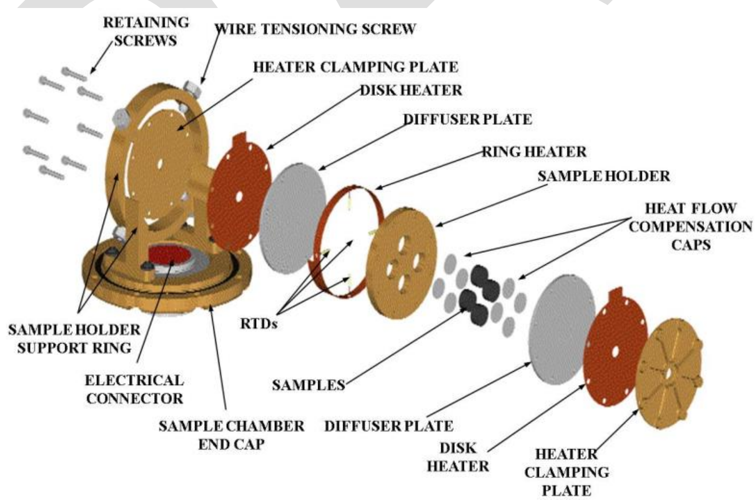


The Coarsening of Solid-Liquid Mixtures's Sample Processing Unit and Electronics Control Unit mounted inside the Microgravity Science Glovebox on ISS Expedition 7.

The SPU houses the four samples that are to be processed. In order to reduce the convective heat transfer, the SPU can be evacuated to approximately 1 x 10E-6 Torr on the ground prior to launch as well as drawing down the pressure just before processing using the ISS Vacuum Exhaust System. The SPU consists of resistive heaters and RTDs to monitor the temperature and provide closed-loop control signals back to the ECU. The sample holder is designed to hold the lead/tin ingot. Each location is exactly measured to provide the PI with the information required to size their samples such that when heated to maximum temperature, they encompass no more than the available volume.



Coarsening of Solid-Liquid Mixture’s Sample Processing Unit sample chamber interior.



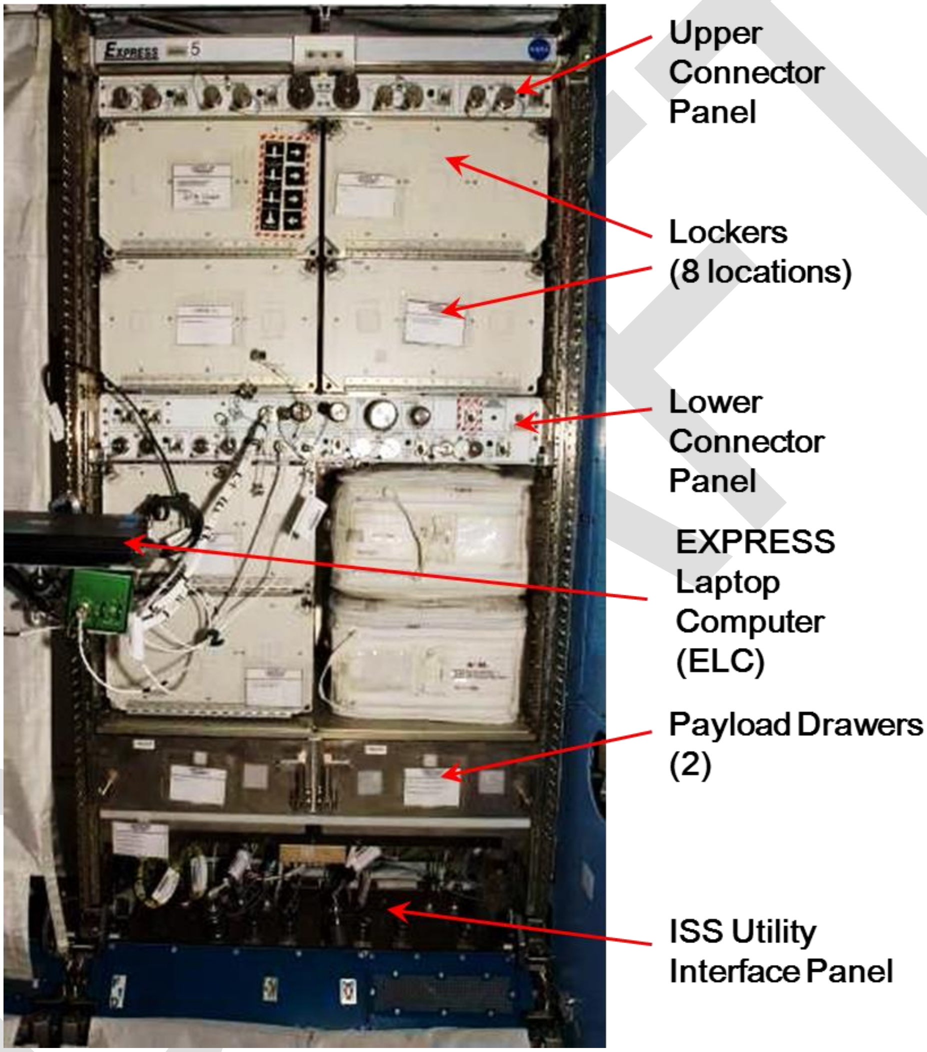
Coarsening of Solid-Liquid Mixture’s Sample Processing Unit sample holder.

Figure: Both the Migrogravity Science Glovebox and the Coarsening of Solid-Liquid Mixtures hardware are in use aboard the ISS and each is available to support additional materials science investigations.

EXPEDITE THE PROCESSING OF EXPERIMENTS TO SPACE STATION (EXPRESS) RACKS

Eight EXPRESS Rack facilities within the ISS laboratories provide standard resources and interfaces for the simultaneous and independent operation of multiple experiments within each rack. Currently, five EXPRESS Racks are located in the U.S. Laboratory, two in the Japanese Experiment Module, and one in the ESA Laboratory.

Each EXPRESS Rack provides eight locker locations and two drawer locations for powered experiment equipment (payloads). Payload developers may use NASA-provided lockers or provide their own structure to occupy the equivalent volume of one, two or four lockers as a single unit.



Expedite The Processing Of Experiments to Space Station Rack configuration.

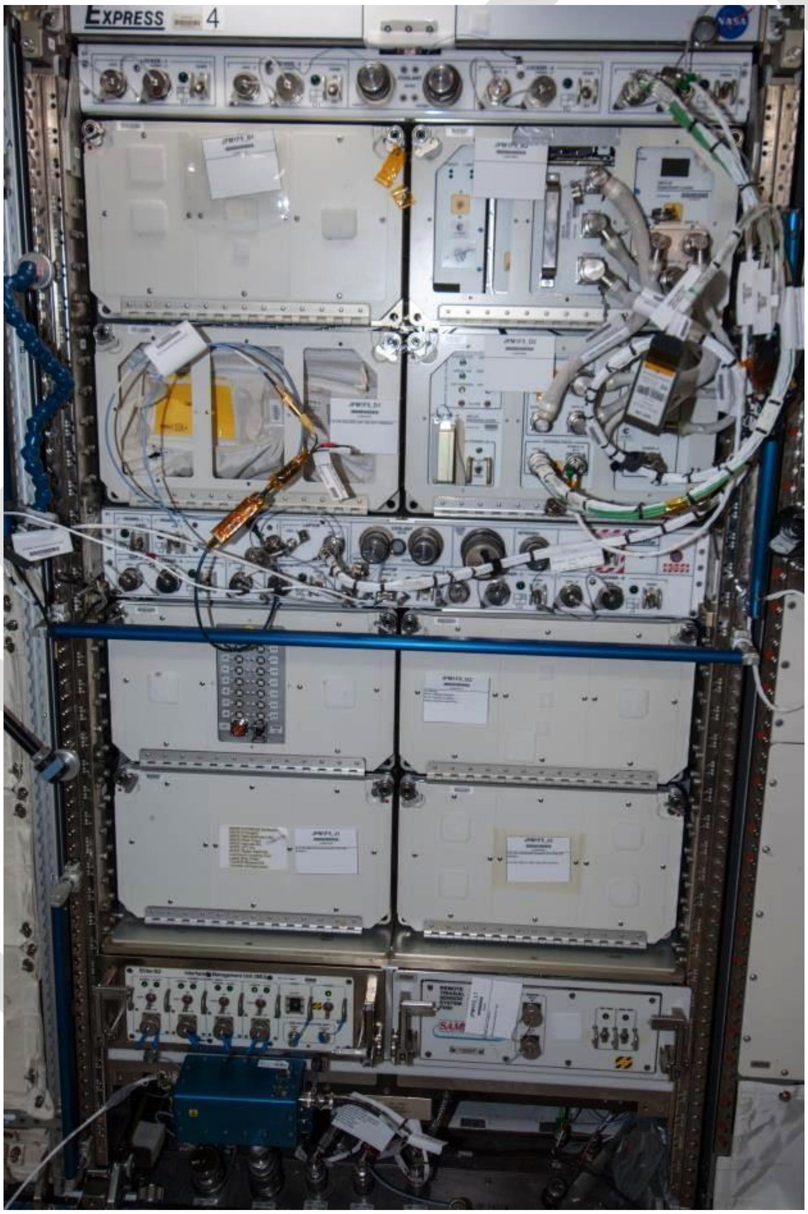
Resources provided for each locker or drawer location include:

- Power (28 Vdc, 0-500 W).
- Command and data handling (Ethernet, RS-422, 5 Vdc discrete, +/- 5 Vdc analog).
- Video (NTSC/RS 170A).
- Rear air cooling (0-200 W).

The Ethernet bandwidth, presently 10 Mbps, is planned to be upgraded to 100 Mbps in 2015. Each rack also provides water cooling (coldplates) from the ISS moderate temperature loop for two payload locations (500 W each), one vacuum exhaust interface and one gaseous nitrogen interface. Standard interfacing cables and hoses are provided in orbit. One Windows-based laptop computer is provided with each EXPRESS Rack to control the rack and to accommodate payload application software. Four of the racks can be equipped with the Active Rack Isolation System to reduce vibration between the ISS and the rack.

NASA provides EXPRESS Rack simulator software for payload developers to develop and checkout payload command and data handling at the developer's site before integrating the payload with the EXPRESS Functional Checkout Unit at MSFC for an end-to-end test before flight.

EXPRESS Racks began supporting investigations aboard ISS on April 24, 2001, and will continue to be available for science experiments through the life of ISS.



EXPRESS Rack 4 with Dispositif pour l'Etude de la Croissance et des Liquide Critiques and NanoRacks payloads, July 2013.

EXAMPLES OF PREVIOUS MICROGRAVITY MATERIALS RESEARCH

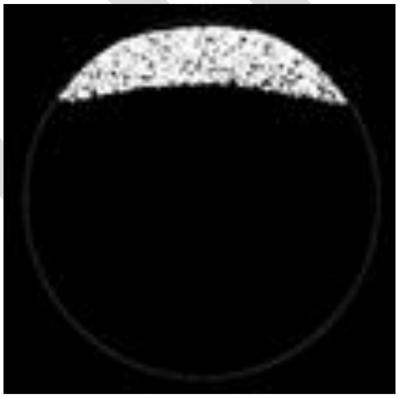
Microgravity materials science research began during the Apollo Program with directional solidification experiments involving semiconductor materials. In Apollo 14, a small, low-temperature furnace was used to cast or directionally solidify a number of two- and three-phase systems. It was found that the dispersion of the phases was much more uniform because of the lack of sedimentation. More sophisticated experiments were conducted on Skylab involving directional solidification of semi-conductors crystals and eutectic materials, brazing, containerless processing to form spheres, etc.

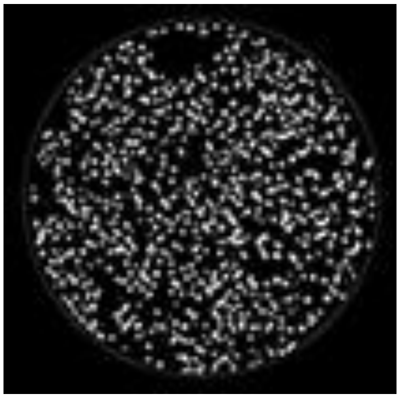
This research continued during the Space Shuttle Program. One notable experiment from the early shuttle flights involved the creation of the first commercial material formed in space. Spherical standards were created from an emulsion of styrene monomers. This emulsion was unstable under normal 1-g conditions because of sedimentation but was quite stable in microgravity. The emulsified styrene monomers were polymerized in orbit to form highly uniform spheres. See Vanderhoff, et al. J. Dispersion Sci. Technology (1984), 5, 231-246.

Microgravity materials research has now moved into the ISS era. Some initial experiments have been performed already and are briefly described below.

COARSENING IN SOLID-LIQUID MIXTURES – 2 (CSLM-2)

This experiment was a follow-on from space shuttle experiments. The ISS experiments were conducted within the sealed Microgravity Sciences Glovebox work volume during Increment 16 in December 2007 and Increment 17 in April 2008. The CSLM experiment was a materials science spaceflight investigation whose purpose was to investigate the kinetics of competitive growth (coarsening) between solid particles essentially suspended within a liquid matrix. CSLM-2 utilized solid particles of tin within a lead-tin liquid. By conducting this experiment in a microgravity environment, detrimental buoyancy and convection effects that arise on Earth were minimized as illustrated in the figures below. .



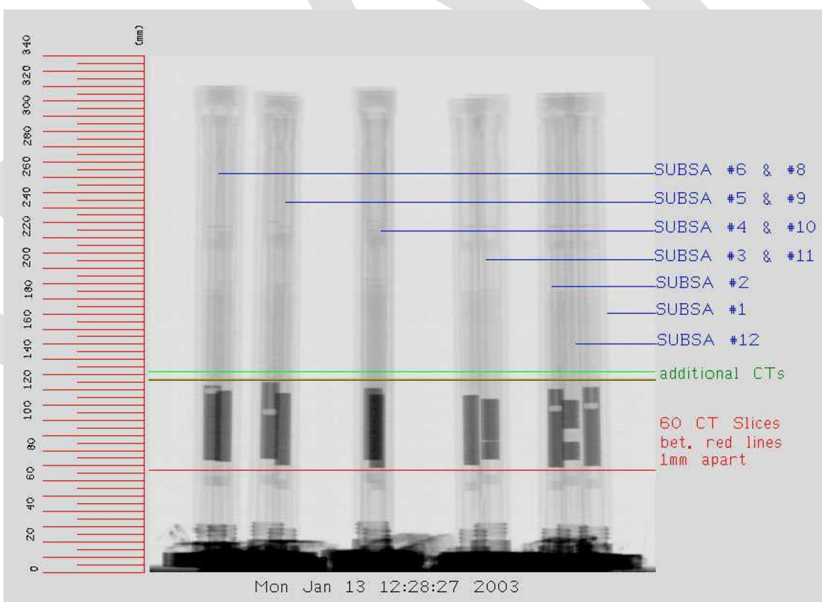


The figure on the right, from an Earth-based experiment, shows an agglomeration of tin particles (white) that rose to the surface because they are lighter than the host lead-tin liquid. On the right, with buoyancy forces minimized in a microgravity environment, a reasonably uniform dispersion of tin particles in the liquid was maintained.

The CSLM-2 experiments were conducted inside the SPU, which has a large, cylindrical chamber. After processing for a given time and temperature, the sample was rapidly cooled to preserve the coarsened structure for later examination on Earth. Six high-volume fraction (tin) samples were successfully completed and returned to Earth and are being evaluated by the Principle Investigator. Initial results unambiguously showed that coarsening occurred by transient Ostwald ripening and the absence of gravitationally induced buoyancy allowed measurements of steady-state coarsening kinetics with unprecedented accuracy. An example of the results was provided in the publication by D.J. Rowenhorst, J.P. Kuang, K. Thornton and P.W. Voorhees, “Three-dimensional analysis of particle coarsening in high volume fraction solid-liquid mixtures,” Acta Mater. 54, 2027-2039 (2006).

SOLIDIFICATION USING A BAFFLE IN SEALED AMPOULES (SUBSA)

The SUBSA investigation was conducted in the MSG aboard the ISS. SUBSA was launched aboard the space shuttle on STS-111/UF-2 on June 5, 2002. Operations were conducted during the Expedition 5 Increment ISS mission. Sample processing occurred between July 10, 2002, and Sept. 11, 2002. The SUBSA samples were returned to Earth by the space shuttle on STS-113, which landed at NASA’s Kennedy Space Center (KSC) on Dec. 7, 2002. The primary science objectives were to visualize the melt/encapsulant behavior in microgravity and to grow improved indium antimonide (InSb) crystals.



Postflight x-ray images of 11 SUBSA flight ampoules taken at the Computed Tomography Facility at NASA/KSC.

Data obtained from the microgravity SUBSA experiments will advance the field of solidification and the technology of crystal growth and thus facilitate growth of more homogeneous and perfect semiconductor crystals on Earth and in space. Specifically, SUBSA data yield values of diffusion coefficients of dopants, which are indispensable for calculations and modeling needed to optimize production of InSb and the data demonstrate the advantages of growing ternary crystals in space, with segregation coefficients k>1. This research was presented in a publication by Alexei Churilov and

Aleksander Ostrogorsky, "Solidification of Te and Zn doped InSb in space", AIAA 2004-1309, 42nd AIAA Aerospace Sciences Meeting & Exhibit, January 2004/Reno, Nevada.

TOWARD UNDERSTANDING PORE FORMATION AND MOBILITY DURING CONTROLLED DIRECTIONAL SOLIDIFICATION IN A MICROGRAVITY ENVIRONMENT INVESTIGATION (PFMI)

The PFMI was conducted in the MSG aboard the ISS. It was selected by the NASA Glovebox Investigation Panel and assigned to the Glovebox Program Office at MSFC on Dec. 3, 1997. PFMI was launched aboard the space shuttle on STS-111/UF-2 on June 5, 2002. The first PFMI experiment was initiated on Sept. 19, 2002. Operations were conducted during the Increment 5, 7 and 8 ISS missions. The PFMI study aimed at understanding porosity formation, its mobility, and effects on structure during controlled directional solidification in a microgravity environment utilizing a transparent material, succinonitrile.

ISS013E14531

Astronaut Jeff Williams changes out tapes during a Pore Formation and Mobility Investigation experiment aboard the International Space Station at the Microgravity Science Glovebox.

Seventeen successful PFMI experiments have been conducted within the microgravity environment of the Materials Science Glovebox provided aboard the International Space Station. A number of results based on PFMI experiments have been obtained, which include agreement with theory on bubble velocity that is due to thermocapillary convection, the first qualitative/quantitative evaluation of aligned gas-solid “eutectic” growth, quantitative assessment of planar interface breakdown in a diffusive environment, and observation of convection in the bulk liquid induced by large bubbles at the interface. The results have advanced the Materials Science field by supplying fundamental data, free of convective effects, for models and theory and by promoting knowledge of bubble behavior in low-gravity environments and their influence on microstructural development. An example of this research was provided by R. N. Grugel, A.V. Anilkumar, and C.P. Lee: "Direct Observation of Pore Formation and bubble mobility during controlled melting and re-solidification in microgravity", Solidification Processes and Microstructures: A Symposium in Honor of Wilfried Kurz, eds. M. Rappaz, C. Beckermann, and R. Trivedi, The Metallurgical Society, Warrendale, PA, 2004, pp. 111-116.

IN-SPACE SOLDERING INVESTIGATION (ISSI)

A proposal entitled the ISSI was submitted in April 2003 and added to the Increment 7 schedule on May 1, 2003. The In-Space Soldering Investigation took place over four increments aboard the ISS in the Maintenance Work Area. Ed Lu (Increment 7) prepared the test coupons from the wire stock and Mike Foale (Increment 8) wrapped the wire pieces with solder. Mike Fincke (Increment 9) conducted the first three sets of soldering experiments, the initial one on July 10, 2004. Leroy Chaio (Increment 10) conducted the remaining two sets, the last on Dec. 22, 2004. The samples were returned on Sept. 23, 2005. The investigation took place as “Saturday Science” with the intent of the experiments to look at joining techniques, shape equilibrium, wetting phenomena, and microstructural development in a microgravity environment. Altogether 28 sets comprising 84 simple and repeatable soldering experiments were conducted aboard the ISS. Basically, the experiments entailed conductively melting either 5- or 10-cm lengths of stock coiled solder onto a test wire that was heated by a soldering iron. The lengths of solder were either initially wrapped around the test wire or fed onto the heated wire.

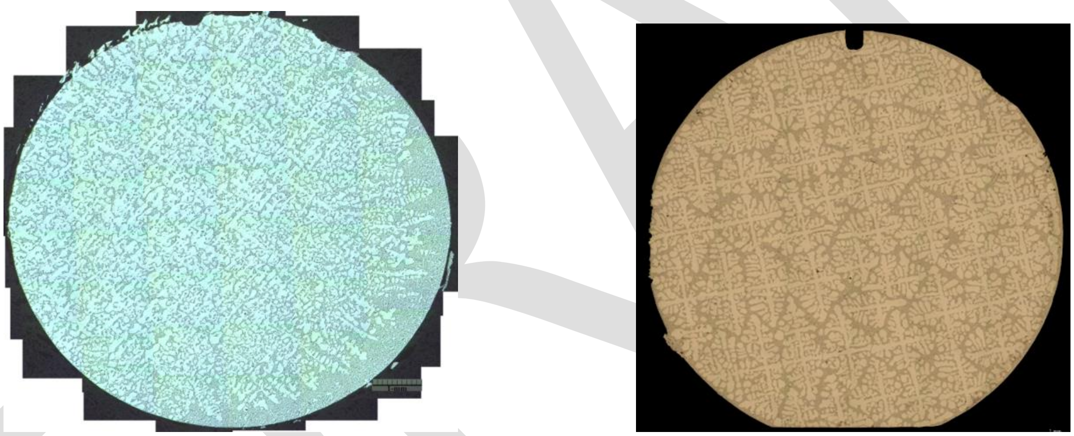
ISS Science Officer Mike Fincke (left) conducts a set of melting experiments within the Maintenance Work Area. The molten solder forms an equilibrium “football” shape around the wire while the ground-based equivalent (right) barely hangs on the wire.

Results from the ISSI study include demonstrating that soldering experiments performed aboard the International Space Station were considerably different than their ground-based counterparts. This was due to Earth’s natural convective flow and buoyancy effects being minimized during melting and solidification, direct observation of gravity-independent thermocapillary flow induced by internal temperature gradients within the molten solder ball, and that in microgravity the internally trapped flux is concentrated at a repair joint that is detrimental to the desired strength and thermal/electrical conductivity. The results were published by R.N. Grugel, L.J. Cotton, P.N. Segrè, J.A. Ogle, G. Funkhouser, F. Parris, L. Murphy, D. Gillies, F. Hua, and A.V. Anilkumar: “The In-Space Soldering Investigation (ISSI):

Melting and Solidification Experiments Aboard the International Space Station," 44th AIAA Aerospace Sciences Meeting and Exhibit, AIAA 2006-521.

COMPARISON OF STRUCTURE AND SEGREGATION IN ALLOYS DIRECTIONALLY SOLIDIFIED IN TERRESTRIAL AND MICROGRAVITY ENVIRONMENTS (CSS)

The CSS investigation is a collaborative effort with the European "Microstructure Formation in Castings of Technical Alloys under Diffusive and Magnetically Controlled Convective Conditions" Investigation. The CSS experiment is performed in the Materials Science Research Rack. The purpose is to determine microstructural development and provide insight regarding defect generation in directionally solidified dendritic metal alloys. The first U.S. sample was processed aboard the ISS in the Low Gradient Furnace on the MSRR/MSL in February 2010; the second sample was processed in January 2011, this time in the Solidification with Quench Furnace module. Both samples have been returned and are currently being evaluated. These samples are characterized by an internal, forest-like, network of metallic branches that directly influence desired material properties.



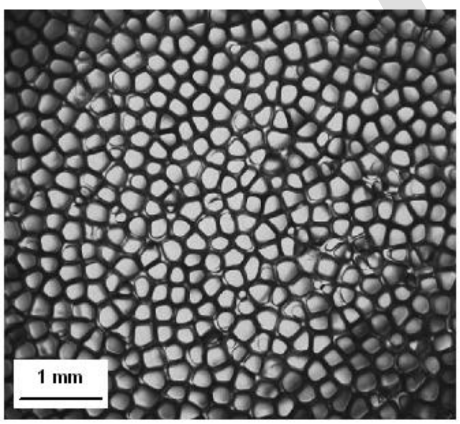
Left: The aluminum – 7wt. percent Silicon sample solidified on Earth exhibits dendrite clustering because of gravity-induced convection. Right: The sample grown in the quiescent microgravity environment of space exhibits a uniformly spaced dendritic network.

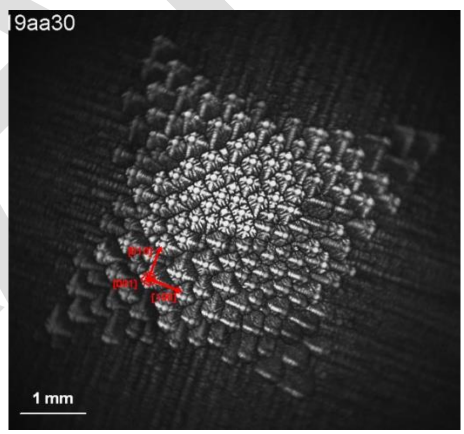
The presence of Earth's gravity induces buoyancy and convective effects during solidification, which disrupt the developing structure and compromises material properties. Solidification experiments in microgravity are strictly diffusion controlled, which promotes a uniform microstructure and leads to improved material properties. The science to be gained is relevant to the technology of directional solidification castings that are used in gas turbine "jet" engines. Evaluation of the microgravity results is currently taking place.

DYNAMIC SELECTION OF 3-D INTERFACE PATTERNS IN DIRECTIONAL SOLIDIFICATION (DSI)

The DSI investigation is a joint effort between the U.S. and the French Space Agency (CNES). The solidification experiments take place in the Directional Solidification Insert of the Dispositif pour l'Etude de la Croissance et des Liquide Critiques (DECLIC) facility aboard the International Space Station and were conducted between April 2010 and May 2011. The objective is to obtain benchmark data required for establishing the detailed dynamics of interface pattern selection during the solidification of alloys.

This research is relevant as understanding of the relationship between processing conditions and resultant microstructure would allow the tailoring of materials to obtain desired properties such as strength and toughness. To achieve these goals, a transparent alloy of succinonitrile is melted and solidified multiple times at different solidification rates through different temperature gradients. Optical observations via microscopy and interferometry are used to record the resultant microstructure. Step changes in velocity are used to establish the role of solidification dynamics on the selection of patterns. Evaluation of the microgravity results is currently taking place.





The micrograph on the left shows the cellular structure that develops in a succinonitrile-camphor alloy solidified at 30µm/s through a temperature gradient of 12°C/cm. A dendritic structure, right, results when the growth velocity is increased. Cellular and dendritic growth and their transitions have been observed and recorded during experiments aboard the ISS. Initial evaluation studies have revealed oscillatory behavior in the diameters of cellular structures under certain conditions.

FUTURE ISS MATERIALS SCIENCE RESEARCH

INTRODUCTION

The NASA Materials Science Program currently has 15 flight investigations that will rely on a variety of ISS research facilities. There are additional investigators who provide numerical modeling to flight experiment teams. With the exception of the CSLM Micro-Gravity Science Glovebox experiments, all the materials flight investigations rely on ISS facilities provided by foreign partners, and a number of these investigations are being performed as part of larger research efforts by international science teams. The current ISS materials science investigations are to be performed between 2013 and 2018. Additional investigations will be selected via a peer review process as these initial investigations are completed. Brief descriptions of the planned investigations are provided.

SEMICONDUCTOR DIRECTIONAL SOLIDIFICATION EXPERIMENTS

Two flight experiments and one modeling effort are being supported by NASA in the field of semiconductor research. The experiments involve directional solidification of a molten sample at slow growth rates, allowing a single crystal to be formed. These flight experiments will be performed in the MSRR.

Reduction of Defects in Germanium-Silicon

This experiment will investigate the quality of germanium-silicon alloy semiconductors solidified from a molten liquid by a process called detached growth. In detached growth, the liquid-solid boundary is detached or not in contact with the wall of the sample container. This condition is difficult to achieve on Earth since gravity tends to press the liquid against the container walls. The method is believed to be capable of producing high-quality semiconductor crystals since defects often form when the liquid solidifies in contact with the surface of the container wall; but relatively little research has been conducted to date.

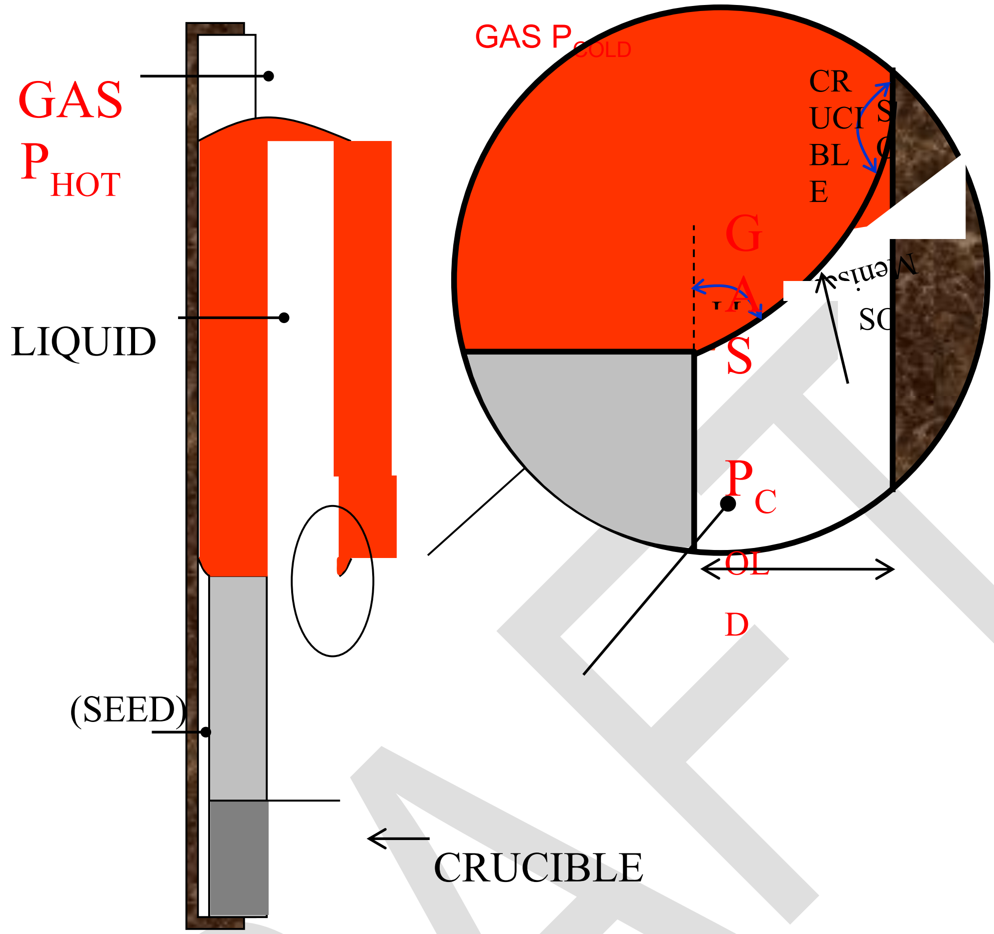


Figure 7: Diagram of detached growth geometry

Crystal Growth of Ternary Semiconductors

The flight experiment will grow (1) crystals of ZnSe-related ternary semiconductors, such as ZnSeTe, ZnSeS, and ZnCdSe, from the vapor phase, and (2) crystals of CdZnTe semiconductor from the liquid phase. The process of crystal growth from a fluid phase on Earth usually introduces density-gradient driven convection in the fluid. Reduction in such convection in low gravity is expected to yield a nearly diffusion-limited growth condition, which results in more uniform growth rates on the microscopic scale and hence greater crystalline perfection and compositional homogeneity. This reduction of convective contamination in a reduced gravity environment will simplify the coupled mass transport and growth kinetics problem. An improved comparison between the experimental results and theoretical simulations is expected and will be beneficial to any growth process on Earth involving mass transport in fluid or fluid-crystal interface growth kinetics.

Modeling of Particle Transport in the Melt and its Interaction with the Solid-Liquid Interface

This project provides modeling support to an international team studying the capture and incorporation of impurities into silicon during solidification. For most semiconductor applications, silicon must be exceptionally pure. Manufacturing costs could be greatly reduced if less pure silicon could be used. This study attempts to understand under what circumstances unwanted impurities in the molten silicon are captured in the solid as it forms causing defects.

Spaceflight experiments will provide tests for the models of the process without the complications of buoyancy effects and fluid flows.

DIRECTIONALLY SOLIDIFIED ALLOYS

Six flight investigations involve the directional solidification of metal alloy samples. An additional two investigations involve modeling of directional solidification of metallic alloys in support of flight experiment teams. One directional solidification flight experiment is conducted in the CNES DECLIC facility. The other flight experiments utilize the MSRR.

Dynamical Selection of Three-Dimensional Interfacial Patterns in Directional Solidification

This series of directional solidification experiments continues the research performed in the CNES built DECLIC facility's Directional Solidification Insert (DSI). The facility utilizes transparent materials to model metallic alloy systems, allowing investigators to observe the evolution of patterns at the solid-liquid interface as they develop. Observations of cellular and dendritic growth and transitions between these growth patterns are to be observed. Initial ISS observations using this facility have already been conducted and interesting oscillatory behavior in the diameters of adjacent cellular structures were observed. A fundamental understanding of cellular pattern formation under diffusive growth conditions is to be developed by comparison of model simulations with benchmark data obtained from the DSI investigation.

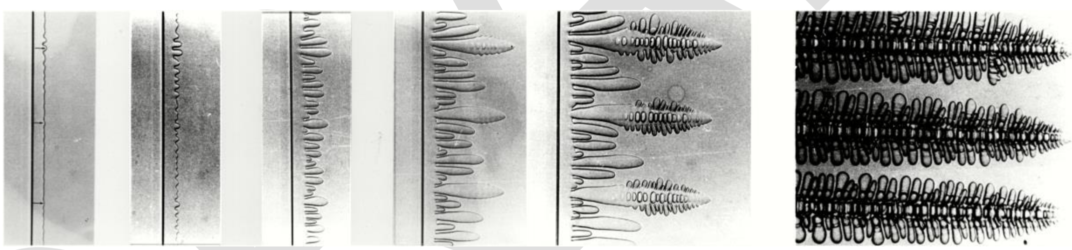


Figure 8: Using a transparent material, it can be observed that as the rate of solidification is increased, the initially smooth liquid to solid interface develops short cells, which lengthen to longer columns, and then transition to increasingly branched dendrites. This is a typical behavior seen in many alloys.

Effect of Convection on Columnar-To-Equiaxed Transition in Alloy Solidification

The objective of the research is to develop computational models of the columnar-to-equiaxed transition (CET) in alloy solidification. This transition occurs as the structure of the solidifying material shifts from finger-like columns to branched, multi-armed structures as the rate of solidification is increased. The resultant properties of the material are largely dependent on whether it forms columnar or equiaxed structures during casting. Gaining a more complete understanding of the complex physical phenomena accompanying the CET is important for predicting the grain structure of castings. Open scientific questions include the role played by melt convection, fragmentation of dendrite arms, and the transport of fragments and equiaxed crystals in the melt. The microgravity experiments provide benchmark data for the case where melt convection and solid transport are absent.

Formation of Gasarites

Materials with gas voids are typically less expensive per unit volume than equivalent solid material. If the voids align then the material can have strength surprisingly close to that of the completely solid material in the direction that the voids are aligned. Voids can be created by dissolving gas in a molten metal, which is liberated as the metal solidifies leaving voids in the solid metal. Materials created by this process are sometimes referred to as gasarites. Alignment of the voids can be difficult to achieve and the buoyancy of the gas likely has a role. In this investigation, metals with aligned voids are to be created on ISS. The effects of varying the solidification rate and the amount of gas dissolved in the metal will be investigated as models predict these are important parameters for describing the process. Results will be used to validate the quality of these models and possibly improve upon them.

3-D Structures and Interface Dynamics Univariant and Invariant Eutectic Solidification in a Ternary Alloy

This work is to develop a 3-D quantitative model of the multiphase alloy microstructures, involving geometric and crystallographic parameters for phase boundaries, domain boundaries (faults), grain boundaries, and the solid-liquid interface itself, and to employ the model to investigate the dynamics of pattern formation during solidification of ternary alloys, i.e., alloys with three types of atoms. Gradient zone directional solidification experiments with no off-axis thermal gradient, off-axis gradient bias, switching gradients, and rotation will be performed on the ground. Univariant (the content of one of the three components varying during solidification) and invariant compositions will be studied. Spaceflight experiments will assist in validation of the model.

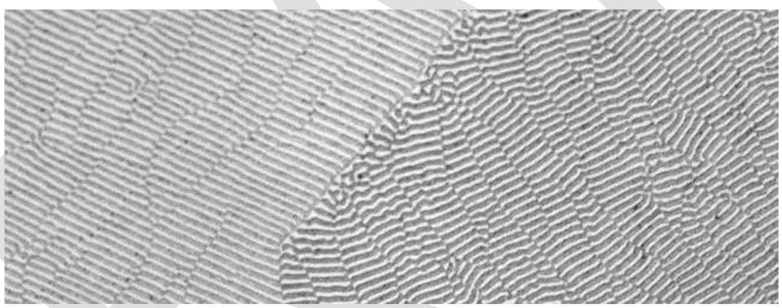


Figure 9: Microscopic image of a grain boundary between a relatively disordered grain on the right and an ordered grain on the left. During the solidification process, the ordered grain grows at the expense of the adjacent, disordered grain.

Effect of Varying Convection on Dendrite Morphology and Macrosegregation

In commercial metal castings, geometries invariably have changes in cross-sections. Yet convection at a cross-sectional change and its relationship to the formation of defects in castings has not been studied adequately. This research comprises the following: 1) directional solidification experiments involving an alloy having a solute that decreases the density of the liquid during solidification (Cu-Sn), an alloy having a solute that increases the density (Al-Cu), and an alloy in which the solute is almost density neutral (Al-Si); 2) crucible designs with variable cross-sections; 3) sophisticated analytical characterizations of the solidified microstructures, including both micro- and macro-segregation; and 4) supporting computer simulations.

Formation of Amorphous Metals In Space

Materials known as bulk metallic glass matrix composites (BMGMCs) are the subject of the investigation. A bulk metallic glass (BMG) is a metal alloy that has been cooled so fast that the atoms within do not have enough time to align in a regular array, imparting a random (amorphous) microstructure to the cooled alloy. This disordered "glassy" state leads to extremely high strengths but brittle failure. By tailoring chemical composition, second phase particles can be grown in the BMG, creating a BMGMC with benchmark combinations of strength and toughness that is due to the presence of these soft particles. Although mechanical properties can be greatly improved in the composites, conventional metal processing is difficult because of issues of second phase growth, viscosity, and sedimentation of the heavier phase. Flight experiments will be used to study the growth of the reinforcing phases in BMGMCs in the absence of sedimentation effects. Success will result in new commercial manufacturing strategies for fabricating hardware from these novel materials.

Integrated Computational and Experimental Studies of Complex Dendritic Microstructure Development during Directional Solidification of Metallic Alloys

This project supports a team performing studies of the formation of columnar and dendritic structures. The aim of the project is to model the formation of dendritic structures in polycrystalline materials where interactions between dendrites in the same grain and grains of different crystal orientation are relevant. The model will incorporate long-range diffusive interactions between primary, secondary, and higher order dendrite branches through a new, coarse-grained “dendritic network” approach. The model will be validated using ground and ISS experiments.

Modeling Peritectic Microstructure Formation during Directional Solidification in Space and on Earth

A peritectic material is one that is formed when a uniform molten liquid is cooled such that initially a solid of one composition forms leaving the liquid with a different composition and then on further cooling the solid and liquid combine to form a solid alloy of uniform composition. Formation of a uniform peritectic alloy is difficult since the liquid composition changes during solidification. This causes density gradients, fluid flows, and non-uniformities in the final composition. This project provides modeling support to an investigation team performing ISS experiments on the formation of a peritectic alloy. The resulting samples should have novel properties for study and provide a test of models of the peritectic process.

ISOTHERMALLY PROCESSED ALLOYS

Two current materials investigations process multi-phase, liquid-solid samples at isothermal conditions. The structures evolve as material from the liquid phase is exchanged with material in the solid phase. This exchange of material occurs through diffusion at the boundary between the phases.

Gravitational Effects in Distortion in Sintering

This experiment involves the sintering process. In the sintering process, cinder-like material is dispersed in a powder consisting of a lower-melting temperature alloy. During processing, the mix is heated until the low-melting temperature material is liquefied. The liquid wets the solid, and material is exchanged between the two phases. The microgravity experiment will allow sintered material to be formed without the complications of fluid flows or sedimentation of the solid particles in the liquid. A number of samples with different compositions and sintering times will be created in order to develop theoretical models that relate the sintering conditions to the final product.

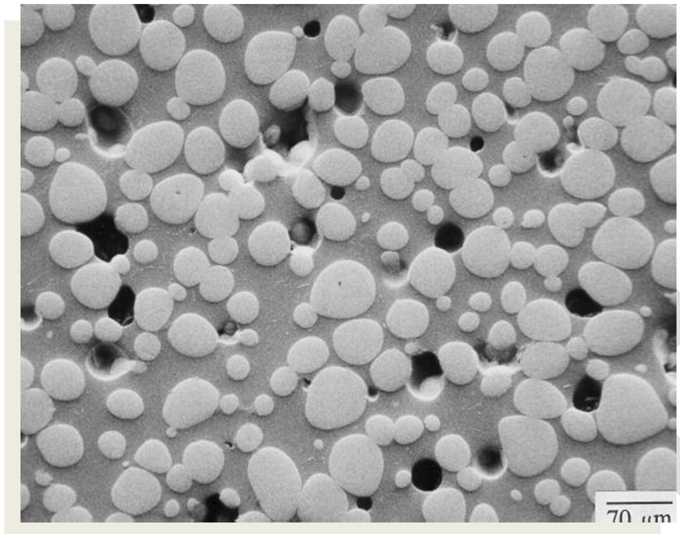
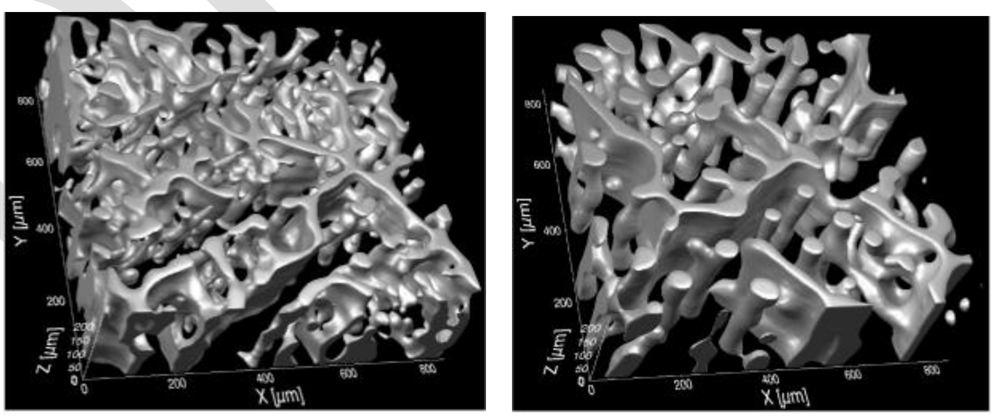


Figure 10: The distribution of phases in a sintered material. The dark areas are pores (voids), the light are the high-temperature material, and the intermediate material is the low-temperature melting material.

Effect of Varying Convection on Dendrite Morphology and Macrosegregation

The investigation concerns the coarsening behavior of a solid dendritic matrix immersed in a liquid metal phase. It is conducted in the ISS Micro-Gravity Science Glovebox using the CSLM hardware. The material in the liquid and solid phases will exchange over time, altering the shape and structure of the dendrites. The flight experiment will involve a low-volume fraction of solid, conditions that usually lead to significant fluids flows and sedimentation of dendrites in the liquid. Samples of different volume fraction are coarsened for varying times and quickly solidified to freeze the dendritic morphology in place at the planned time in the coarsening process. A series of cross sections are cut through the samples, and analytical techniques are then used to characterize the dendritic structures. The resultant data are used to improve models of the coarsening process. Initial experiments have been performed, and more are planned.



(a) (b)

Figure 11: 3-D reconstructions of the evolution of an Al-Cu alloy after coarsening for (a) 10 minutes and (b) 90 minutes.

THERMOPHYSCIAL PROPERTY MEASUREMENT VIA ELECTRO-MAGNETIC LEVITATION EXPERIMENTS

Five of the NASA materials science investigations are to be performed in the Electro-Magnetic Levitation (EML) facility between 2013 and 2018. The first delivery of EML samples to ISS should occur in 2013 with experiments on the initial samples occurring over the subsequent months. A new set of EML samples is to be delivered to ISS each year through 2018. EML investigations study the properties and behavior of molten alloys as they cool and solidify. The combination of the microgravity and electro-magnetic fields allow the investigators to study the transition from the liquid to the solid state in conditions where the fluid flows are precisely controlled. The levitated sample is floating and not in contact with a container during solidification so the effects of container walls on the solidification process are absent. The absence of container walls frequently results in conditions where deep undercooling of the sample can be obtained. (Undercooling means that the sample remains molten below its normal freezing point.) This occurs because in the absence of a sample container, the liquid has no solid surface to use as a nucleation point for solidification. EML researchers will measure properties of molten and solidifying alloys such as the viscosity, surface tension, rates of solidification, etc. The NASA-supported researchers are involved in the following experiments.

Quasi-Crystalline Undercooled Alloys for Space Investigation (QUASI).

An icosahedral quasi-crystal is an ordered phase not capable of forming a true periodic 3-D structure. It is thought to play a critical role in the formation of metallic glasses, which have exceptional strength and toughness and do not have a regular 3-D order. QUASI seeks to determine the influence of short-range order on the nucleation barrier; correlate local structure in the liquid with nucleation kinetics and with thermophysical properties; and to develop, evaluate, and refine a model for nucleation kinetics. Quasi-crystals have received a great deal of attention recently because of Dan Shechtman, an Israeli scientist who was recently awarded the Nobel Prize in chemistry for his work in discovering quasi-crystals. An example of a Ca-Cd icosahedral quasi-crystal is shown below:

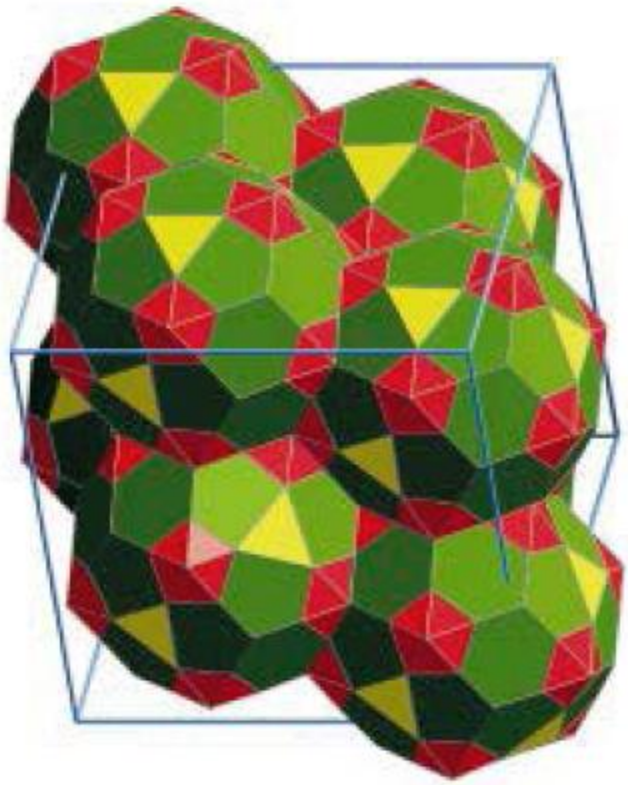


Figure 12: The icosohedral shape of quasi-crystals does not allow them to completely fill a symmetric 3-D space. Gaps remain in places between the individual quasi-crystals.

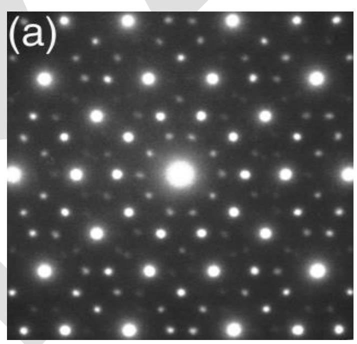


Figure 13: An X-ray diffraction pattern of a Ti37Zr42Ni21 sample shows five-fold pattern indicative of a quasi-crystal.

Thermophysical Properties and Solidification Behavior of Undercooled Ti-Zr-Ni Liquids Showing an Icosahedral Short-Range Order (ICOPROSOL)

ICOPROSOL investigates the nucleation and growth of quasi-crystals and the effect of atomic-scale order on the macroscopic properties of these alloys. One goal of the proposed research is to study the influence of the short-range order in the liquid phase on the nucleation behavior of ordered solid phases of Ti-Zr-Ni. The investigations performed under ICOPROSOL may improve our ability to tailor the microstructure of metals for commercial applications.

Thermolab

Thermolab investigates the thermophysical properties of high-temperature materials, many of which are used commercially. A better understanding of the physical properties will allow more efficient and more reliable production of metallic parts using these alloys. Alloy compositions are chosen based on the scientific interest in their properties. The data obtained from EML may expand the fundamental understanding regarding the formation of bulk metallic glasses (disordered, non-crystalline metals) and quasi-crystals.

Peritectic Alloy Rapid Solidification with Electro-Magnetic Convection (PARSEC)

PARSEC investigates the effect of fluid flow on the solidification path of peritectic structural alloys. These materials initially nucleate to form a metastable phase consisting of a liquid and solid followed by transition to a second solid phase. The final product can exhibit properties very dependent on the convection associated with processing conditions. Understanding this relationship would enable control of the solidification path so that the microstructure and properties of peritectic materials may be tailored for specific applications.

Levitation Observation of Dendrite Evolution in Steel Ternary Alloy Rapid Solidification

The focus of this effort is to study metastable solidification in steel alloys. Many steel alloys consist of two phases, one phase that is thermodynamically stable and another that is metastable. Both may appear in an alloy because even though the metastable phase may not be thermodynamically favored it can be quicker to nucleate and grow. Undercooling in containerless processing has produced metastable phases and materials that exhibit improved chemical homogeneity and ultra-fine grain sizes. By observing changes in the mechanism for nucleation of the stable phase following nucleation of the metastable phase from undercooled melts, the role of convection in phase selection may be evaluated. The results have application to the design of industrial welding, spray forming and strip-casting operations for steels.

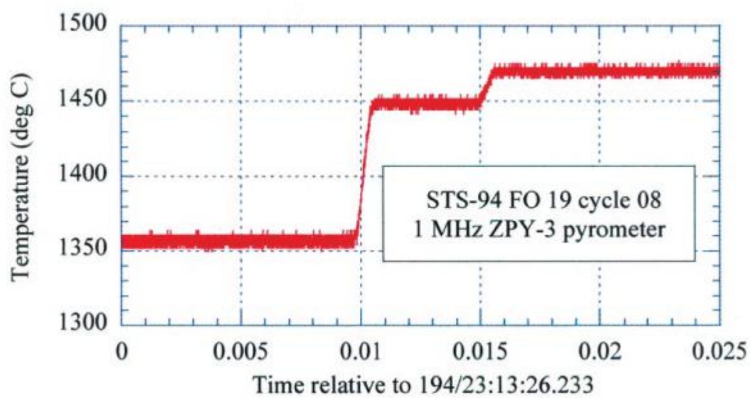


Figure 14: Temperature data taken by an optical pyrometer indicates that the sample undergoes two solidification events. Heat is released increasing the sample temperature as the metastable and then stable phases form from the undercooled liquid.

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ACRONYMS – MICROGRAVITY

BMG	Bulk Metallic Glass
BMGMC	Bulk Metallic Glass Matrix Composites
CET	Columnar-to-equiaxed Transition
CLSM	Coarsening of Solid-Liquid Mixtures
CNES	French Space Agency
CSS	Comparison of Structure and Segregation
DECLIC	Dispositif pour l'Etude de la Croissance et des Liquide Critiques
DSI	Directional Solidification Insert
ECU	Electronics Control Unit
EML	Electro-Magnetic Levitation
ESA	European Space Agency
EXPRESS	Expedite The Processing Of Experiments to Space Station
FI	Furnace Inserts
ICOPROSOL	Icosahedral Short-Range Order
ISS	International Space Station
ISSI	In-Space Soldering Investigation
KSC	NASA's Kennedy Space Center
LGF	Low Gradient Furnace
MSFC	NASA's Marshall Space Flight Center
MSG	Microgravity Science Glovebox
MSL	Materials Science Laboratory
MSRR	Materials Science Research Rack
PARSEC	Peritectic Alloy Rapid Solidification with Electro-Magnetic Convection
PFMI	Pore Formation and Mobility Investigation
PI	Principle Investigator
QUASI	Quasi-Crystalline Undercooled Alloys for Space Investigation
SCA	Sample-Cartridge Assemblies
SPU	Sample Processing Unit
SQF	Solidification and Quenching Furnace
SUBSA	Solidification Using a Baffle in Sealed Ampoules
WV	Work Volume